原　　著

# Titanium Casting: Effect of Mould Condition of Magnesia-based Investment

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## ABSTRACT

Dental casting of pure titanium metal was investigated by mould condition of magnesia-based investment as a dental investment mould. To select setting condition after mixing of the investment, the selected holding temperatures were room temperature (RT), 70°C and 100°C in the oven. After setting, the investment was heated to 900°C and held for 2 hours and subsequently cooled to 500°C or additionally cooled to 100°C for titanium casting. Two types of dental waxes, two types of paraffins and one cast pattern resin were used as a wax pattern. Based on the surface appearance of the investments after setting, five kinds of casting conditions were examined by the surface appearance of titanium crown castings which were using a vacuum-argon pressure casting machine with arc melting. Their mechanical strength and thermal properties of magnesia-based investments which were obtained under different setting conditions were measured by compressive strength and thermal expansion value, thermogravimetry (TG) and differential thermal analysis (DTA). The results showed that two waxes exhibited smooth investment mould surface before the casting. The appropriate wax (dental waxes)/setting condition (RT) combination gave titanium crown castings with macroscopically smooth surface when cast to investment mould at 100°C.

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## INTRODUCTON

Dental magnesia-based and phosphate-bonded investments have been developed for titanium casting, because casting procedures have some problems due to high fusion temperature of titanium metal (1683°C) and the adhesion between investment mould and metal[1-5]. Casting procedures were controlled by casting methods[2-4] (a vacuum-argon pressure casting with arc melting and centrifugal casting with induction melting). The investment mould types affected surface characteristics of cast metal and alloys, because there appeared a thermal reaction between investment mould and metal. The composition of cast investment controlled the value of surface roughness, because the mould temperature and environmental atmosphere affected the mecganical strength and thermal change of investments after setting (preheating) or during heating in the furnace[6-10]. From these observation, experimentally formulated investments were developed to obtain the perfect titanium castings using cast machine with high melting systems[11,12]. Dental magnesia-based investment and phosphate-bonded investment were applied to high fusing metals and alloys[13]. The additives of aluminium and indium pure metals in experimental nickel-based Ni-Cu-Mn ternary alloys were used to minimize the formation of adhesive oxides due to thermal reaction between alloy and investment mould, showing that the adhesive oxides were removed macroscopically and the effective contents of additive metals were below 10 wt%[14].

In this study, six setting conditions of magnesia-based investment after heating to 900°C and subsequently to 500°C were examined by the macroscopic observation of investment mould when five types of waxes and one cast pattern resin were used as a wax pattern to form the cast crown. The setting behaviours and mechanical strength were analysed by thermal properties and fracture strength of cast investment mould, and the macroscopic appearance of titanium crown castings were examined under five heat conditions at selected mould temperatures.

## MATERIALS AND METHODS

### 1. Materials

1.1 Magnesia-based investment

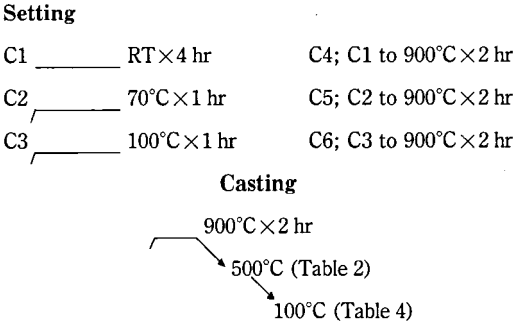
The magnesia-based investment mould was ethyl silicate-bonded magnesia-based investment, which contained 50 wt% magnesia (MgO) and 50 wt% alumina ($Al_2O_3$) powders with very small amount of metal oxide as zirconia, patented by one of the present authours. The magnesia-based investment had silica gel as the binder by means of sol-gel reaction[13]. The mixing liquid including hydrolysed silica sol solution (Colcoat Co, Tokyo) and an ammonium carbonate solution which were adjusted in this study (catalyst; Katayama Kagaku Kogyo, Tokyo). They were used as 16 ml (silica sol)/1.1 ml (catalyst), based on the earlier reports[15,16], and the mixing liquid /investment powder ratio was 0.15. After setting of investment (10 minutes) within the casting ring which was made by acrylic resin (thickness = 1 mm), it was removed to hold it under six setting conditions. The ring had dimension of 34 mm outer diameter and 45 mm height. In this study, so-called ringless casting was carried out under a vacuum-argon pressure casting (see section 3).

1.2 Wax patterns

Four waxes and one cast pattern resin were used as a wax pattern from the same metal crown as the earlier report[13]. Two types of dental waxes for the pattern was a dental wax; Undercut wax (GC Co, Tokyo; 122°C as a melting temperature; W1) and a dental wax (Blue-inlay wax; GC Co, Tokyo; 50°C; W2). Two types of paraffins had different melting temperatures 68～70°C (Wako Junyaku Kougyou, Osaka; W3) and 56～58°C (Katayama Kagaku Kougyou; W4). One cast pattern resin (Nisshin Co, Kyoto; W5) had 50°C as a melting temperature. Immeadiately after making the watterns, they were conserved at room temperature (RT) for 4 hours, 70°C for 1 hour, and 100°C for 1 hour, and they were subsequently heated to 900°C and held for 2 hours (Table 1). The surface porosity was observed macroscopically on the investment surface when C5 condition was used and then cooled to 500°C.

Two heat conditions before casting, 1) a heat to 900°C and a holding for 2 hours and subsequently cooled to 500°C and 2) subsequently holding it in the oven (controlled at 100°C), were examined (Table 1). Examining titanium castings, the latter condition was selected, because the investment samples showed no cracks on the surface which were introduced by cooling. Also, based on macroscopic observation, the wax types were selected. Using three types of waxes, W1, W2, and W3, the titanium casting was done and the surface defect was examined macroscopically.

**Table 1** Schematic diagrams of six setting conditions ( C1,C2, C3, C4, C5, and C6) and two casting methods. For key, see text.




### 2. The properties of invetments investigated

2.1 Compressive strength

The fracture strength value ($4L/3.14\ d^2$; L = load at fracture) was estimated by compressive strength of investment samples (30 mm diameter and 40 mm height) of load/deflection curve which were set under the condition, C1, C2, and C3 (sample size = 5), using a AUTOGRAPH testing machine (DCS type, Shimadzu Co, Kyoto); 100 kgf (full scale), 2 mm/min (cross-head speed), 20 mm/min (chart speed). The strength value ($kg/mm^2$) was converted to SI unit (MPa).

2.2 Thermal expansion

Thermal expansion of magnesia-based investment used was examined by thermal analyzer (Thermoflex, RIGAKU, Tokyo). The heating rate to 800°C was 10°C/min under the degassed atomosphere. The cylindrical specimen dimension was 5 mm diameter and 12 mm height.

Reference sample was a silica glass (5 mm diameter and 12 mm height) and the expansion value was calibrated. Five test samples were used for each case under thermal analyses.

2.3 TG and DTA analyses

Thermogravimetry (TG) and differential thermal analysis (DTA) were carried out using DT-50 (Shimadzu Co, Kyoto). The samples were heated to 900°C with a heating rate of 10°C/min during thermal analyses. The investment sample weight was 20 mg. The TG full scale was 20 mg and DTA full sacle was 200 $\mu$V under six conditions of setting (C1, C2, C3) or heating(C4, C5, C6) (Table 1). The same measurement conditions as earlier reports were used for TG and DTA analyses[15-17].

### 3. Titanium casting

The material tested (15 gram ingot) was a pure titanium metal (KS-50, Kobe Steel Co, Kobe), which contained Fe 0.06 wt% , $H_2$ 0.004 wt%, C 0.007 wt%, $N_2$ 0.01 wt% and $O_2$ 0.14 wt%. The metal samples were cast by vacuum-argon pressure after arc melting which was called to be pressure arc casting (sample size = 5) (Advanced Arc Casting System CYCLARC Hiroshima Daigaku Shigakubu Model I; J. Morita Co, Kyoto; Fig. 1). Argon pressure was 14.71 kPa, and a melting current was 190A during 30 seconds melting time, using pure Cu crusible. The surface observation of titanium crown castings, which were obtained by five setting conditions, was done macroscopically.

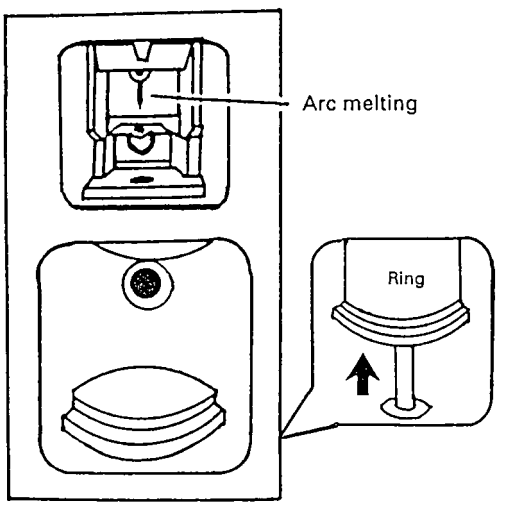



**Figure 1** Casting machine (schematic figure). Originally, it has been already indicated[13].

## RESULTS

Table 2 indicates the macroscopic appearance of magnesia-based inbvestment surface. The specimen samples were obtained by W1 to W5 conditions and then treated by C5 condition and subsequently cooled to 500°C. The appearance of ranking was macroscopically described, showing that better conditions were W1 and W3.

Table 3 indicates compressive strength at fracture of magnesia-based investments which were obtained under C1, C2 and C3 conditions. The samples set under three conditions, showing that the heated samples had a larger strength than that of RT-conserved sample (C1). Figures 2, 3, and 4 show thermal properties of thermal expansion, TG and DTA characteristics, respectively. The thermal expansion had an increased value by adding alumina to magnesia powderin the temperature range of more than 400°C (0.6%). The weight loss which occurred with sol-gel reaction of ethylsilicate sol (liquid part) started at about 300°C and finished at 350°C, as indicated (C1,C2 and C3 conditions), whereas C4,C5 and C6

**Table 2** Macroscopic appearance of magnesia-based investment when treated by W1 to W5 waxes. C5 condition in Table 1 and subsequently cooled to 500°C (see text for key).

| Code | Wax | Appearance |
|---|---|---|
| W1 | Undercut wax (122°C) | +++ |
| W2 | Blue-inlay wax (50°C) | ++ |
| W3 | Paraffin (68~70°C) | +++ |
| W4 | Paraffin (56~58°C) | + |
| W5 | Cast pattern resin (50°C) | − |

+++; smooth investment surfaces
++; porosity partly existed on the surface
+; less porosity than ++
−; small size of porosity existed uniformly over the surface

**Table 3** Compressive strength which was obtained under C1, C2, and C3 conditions. For key, see text.

| Code | Compressive strength (MPa) |
|---|---|
| C1 | 0.74 (0.06) |
| C2 | 1.07 (0.02) |
| C3 | 2.25 (0.05) |

Mean (standard deviation)

**Table 4** Macroscopic apperance of titanium cast crown when hardened under the WA to WE conditions. Setting was carried under RT (C1) or 100°C (C3) condition, and casting was done after holding at 100°C for 10～30 minutes (for key, see text).

| Code | Wax/Setting temperature | Appearance |
|---|---|---|
| WA | W1/100°C | − |
| WB | W1/RT | ++ |
| WC | W3/100°C | − |
| WD | W3/RT | − |
| WE | W2/RT | +++ |

+++; smooth metal surfaces
++; porosity partly existed on the surface
+; less porosity than ++
−; small size of porosity existed uniformly over the surface

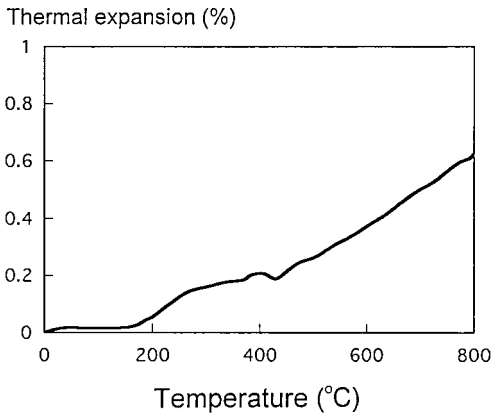



**Figure 2** Thermal expansion of magnesia-based investment.

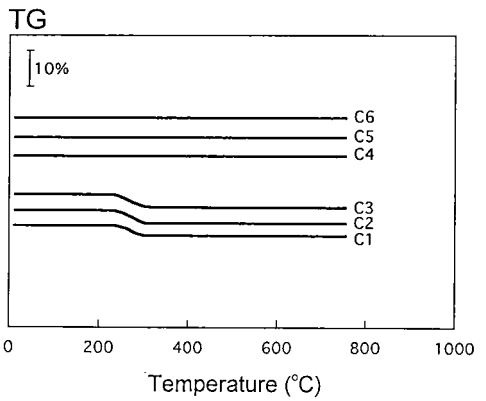



**Figure 3** TG change with test temperature under C1 to C6 conditions. See key for text.

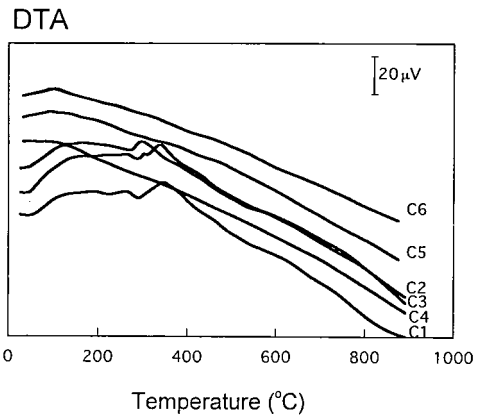



**Figure 4** DTA change with test temperature under C1 to C6 conditions. See key for text.

condition gave no weight loss of investment. These trends corresponded to a peak (300 ～ 350°C) due to exothermic reaction on DTA curves for C1, C2 and C3 conditions. The other conditions had no peak in such temperature range.

Table 4 indicates the same appearance of titanium crown castings as Table 2. The appearance exhibited WB and WE conditions when held at 100°C, because the smooth surface was observed on the cast surface.

## DISCUSSION

The investment type affected the fit of titanium castings[18] and colour stability[19] and surface condition[20]. As a laboratory procedure for titanium crown casting, setting and thermal expansion and casting experiment were very important to clarify the effect of investment on titanium castings. As reported by earlier reports[1−13], silica-based investments and magnesia/alumina-based investments were applied as a investment mould. The relatively low mould temperature recommeded were below 100°C[13] or 200 or 350°C[18], because these investment exhibited heavy reaction with the mould. For the metal compensation of metal shrinkage, zero or negative expansion of investment was needed. Recent study reported that mould reaction occurred little when the investment was heated to 950°C and then cooled to 600°C[18]. Thus, in this study, setting condition of magnesia-based investment with different types of waxes and casting condition when cooled to 500°C or held at 100°C was examined.

The titanium casting was affected by mould filling with different gas permeability[9]. Thus, imvestment types, such as phosphate-bonded silica investment and magnesia-

based investments have been developed experimentally[5,6,14]. To increase castability to obtain complete castings, the development of investment mould was needed. Syverud and Hero reported that an arc casting machine with a separate melting and mould chamber was important because both chambers were evaculated before argon was introduced into melting chambers[9]. Thus, the gases in the mould cavity escaped through the investment to the mold chamber. The magnesia-based investment used in this study had produced complete castings, as reported that it had higher gas permeability than the other commercial magnesia-investments when fired to each recommended temperature and cooled to RT[9]. From their reports, this study exhibited that there appeared no chemical reaction at more than 400°C as indicated by no peak on the curves in Figs 3 (TG) and 4 (DTA). As a setting condition, the magnitude of compressive strength at a conservation at RT was reasonable (Table 3), because the compressive strength for the inlay investments had approximately 2.46 MPa when tested 2 hours after setting[21]. The appropriate heat temperature during heating was 900°C, and also the holding temperature was better at RT than at 100°C (Table 4).

Metal shrinkage was compensated when zero or negative mould expansion, showing that the discrepancy between crown casting and the die was 0.15 mm as a fit of the titanium crown[18]. The magnesia-based (magnesia-alumina) investment showed a better fit to the die using the same mould condition as this study[13]. As a laboratory procedure for titanium crown casting, the relatively low mould temperature was thus needed.

This study clarified that the complete titanium crown castings were achieved by hiring at 900°C for 2 hours after setting at RT, and subsequently cooling to 500°C and then holding at 100°C before casting. This setting and heat conditions suggest that mold filling of magnesia-based investment has high gas permeability during heating and casting. The optimal condition of magnesia-based investment mould was confirmed. In this study the mould conditions of setting and heating were obtained experimentally, and this method followed by a second cooling and holding temperature (100°C) after cooling to 500°C can be recommended for titanium crown castings.

## CONCLUSION

Pure titanium metal was cast under a vacuum-argon pressure casting, examining the setting condition of magnesia-based investment. After setting at RT, or 70°C, or 100°C, the investment mould was heated to 900°C, and then cooled to 500°C or additionally held at 100°C in the oven for titanium casting. Based on the surface appearance of the investments after setting by selecting wax patterns, five kinds of casting conditions were examined by the surface appearance of titanium crown castings. The results showed that two waxes exhibited smooth investment mould surface before the casting. Using paraffin or dental wax/setting condition at RT, titanium crown castings with less porosiy on the cast surface were achieved under the mould condition that was subsequently placed at 100°C for 10～30 minutes after cooling to 500°C.

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